



Relating soil C and organic matter fractions to soil structural stability

Jensen, Johannes L.; Schjønning, Per; Watts, Christopher W.; Christensen, Bent T.; Peltre, Clément; Munkholm, Lars J.

Published in:
Geoderma

DOI:
[10.1016/j.geoderma.2018.10.034](https://doi.org/10.1016/j.geoderma.2018.10.034)

Publication date:
2019

Document version
Publisher's PDF, also known as Version of record

Document license:
[CC BY](#)

Citation for published version (APA):
Jensen, J. L., Schjønning, P., Watts, C. W., Christensen, B. T., Peltre, C., & Munkholm, L. J. (2019). Relating soil C and organic matter fractions to soil structural stability. *Geoderma*, 337, 834-843.
<https://doi.org/10.1016/j.geoderma.2018.10.034>



Relating soil C and organic matter fractions to soil structural stability

Johannes L. Jensen^{a,*}, Per Schjønning^a, Christopher W. Watts^b, Bent T. Christensen^a, Clément Peltre^c, Lars J. Munkholm^a

^a Department of Agroecology, Aarhus University, Blichers Allé 20, 8830 Tjele, Denmark

^b Department of Sustainable Agriculture Sciences, Rothamsted Research, Harpenden, Hertfordshire AL5 2JQ, United Kingdom

^c Department of Plant and Environmental Sciences, University of Copenhagen, Thorvaldsensvej 40, 1821 Frederiksberg, Denmark

ARTICLE INFO

Handling Editor: Morgan Cristine L.S.

Keywords:

Soil structural stability
Soil organic carbon
Permanganate oxidizable carbon
Hot water-extractable carbon
Soil management

ABSTRACT

Soil organic matter (SOM) is important for maintaining soil structural stability (SSS). This study quantified the influence of soil organic carbon (SOC) and different organic matter components on various SSS measures. We used a silt loam soil with a wide range of SOC (8.0–42.7 g kg⁻¹ minerals) sampled in spring 2015 from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research. Four treatments were sampled: Bare fallow, continuous arable rotation, ley-arable rotation, and grass. Soils were tested for clay dispersibility (DispClay), clay-SOM disintegration (DI, the ratio between clay content without and with SOM removal) and dispersion of particles < 20 µm. The SSS tests were related to SOC, permanganate oxidizable carbon (POXC), hot water-extractable carbon (HWC), mid-infrared photoacoustic spectroscopy (FTIR-PAS) and mineral fines/SOC ratio. SSS increased with increasing content of SOM components. The relationships between SOM components and SSS followed a broken-stick regression with a change point at ~23.0 g SOC kg⁻¹ minerals (clay/SOC~10) coinciding with a change from the tilled treatments to the grass treatment. We found a greater influence of SOC, POXC and HWC on SSS at contents below the change point than above. A stronger linear relation between POXC and DispClay compared to SOC and HWC suggests that POXC was a better predictor of the variation in DispClay. POXC and HWC were less related to DI than SOC. The grass treatment had a very stable structure, shown in all SSS tests, probably due to the absence of tillage and large annual inputs of stabilizing agents. This suggests that a change in management from arable rotation to permanent grass is one effective tool for improving SSS.

1. Introduction

The importance of soil organic matter (SOM) on key soil functions is well known (e.g., Johnston et al., 2009), and consequently loss of SOM is considered a major threat to sustained soil functions (Amundson et al., 2015). One soil property affecting key soil functions is soil structure. Soil structure is the relative arrangement of particles and pores (Dexter, 1988), and the ability of soil structure to resist external stresses both mechanical and or from water is termed soil structural stability (SSS). Greater SSS is essential for minimizing the risk of downward transport of fine particles carrying pollutants to the water environment (de Jonge et al., 2004), soil erosion (Le Bissonnais, 1996), soil cementing, and seedbeds with hard and non-friable aggregates (Kay and Munkholm, 2004). SOM content is an important factor affecting

SSS (Bronick and Lal, 2005), and a range of studies have shown that an increase in SOM content increases SSS (e.g., Jensen et al., 2017a; Watts and Dexter, 1997).

Soil organic carbon (SOC) is the main constituent of SOM and may serve as a proxy for SSS. Labile organic compounds are potentially better indicators for soil functions (Haynes, 2005). For example, permanganate oxidizable carbon (POXC) is considered a labile component of SOM and has been found to be more sensitive to differences in management than total SOC (Culman et al., 2012). POXC is an easy and low-cost method and POXC has been suggested the best single predictor of soil health (Fine et al., 2017) and a better predictor of crop productivity than total SOC (Hurisso et al., 2016). Similarly, hot water-extractable carbon (HWC) is highlighted as a soil quality indicator more sensitive to management changes than total SOC (Ghani et al., 2003).

Abbreviation: A, Continuous arable rotation; BF, Bare fallow; CEC, Cation exchange capacity; DI, Clay-SOM disintegration; DispClay, Clay dispersibility; DispFines20, Dispersion of particles < 20 µm; Fines20, Mineral particles < 20 µm; FTIR-PAS, Mid-infrared photoacoustic spectroscopy; G, Grass; HWC, Hot water-extractable carbon; LA, Ley-arable rotation; LF-free-SOC, Light fraction-free-SOC; LFSOC, Light fraction organic carbon; NTU, Nephelometric turbidity unit; PCA, Principal component analysis; POXC, Permanganate oxidizable carbon; SSA, Specific surface area; SSS, Soil structural stability

* Corresponding author.

E-mail address: jlj@agro.au.dk (J.L. Jensen).

<https://doi.org/10.1016/j.geoderma.2018.10.034>

Received 18 January 2018; Received in revised form 18 May 2018; Accepted 19 October 2018

Available online 03 November 2018

0016-7061/ © 2018 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

HWC is considered a labile component of SOM consisting of microbial and plant derived material (Hirsch et al., 2017; Villada et al., 2016). Another measure, which potentially could better explain changes in SSS than total SOC, is light fraction (LF) free SOM. This can be obtained by subtracting light fraction organic carbon (LFSOC) from total SOC since LFSOC is a fraction not closely associated with mineral particles (Gregorich et al., 2006). Finally, mid-infrared photoacoustic spectroscopy (FTIR-PAS) can be used to assess differences in SOM quality (Peltre et al., 2014; Peltre et al., 2017), which potentially could improve the explanatory power in predicting SSS.

Increasing evidence suggest that soils exhibit a capacity factor for SOC stabilization also known as saturation state (Hassink, 1997; McNally et al., 2017). The saturation state of the soil has been found to influence the SSS measure, clay dispersibility, more than SOC per se (Dexter et al., 2008). The saturation state can be expressed by the clay/SOC ratio. A critical value close to 10, corresponding to a soil where the mineral particles are considered to be saturated with SOC, has been found in several studies (Dexter et al., 2008; Getahun et al., 2016; Jensen et al., 2017a; Schjønning et al., 2012). Soils with clay/SOC above 10 are considered SOC-unsaturated, and for such soils, SSS may be reduced. A corresponding threshold has been found for mineral particles < 20 µm (Fines20) where the ratio of Fines20/SOC above 20 indicates reduced SSS. Consequently, these mineral fines to SOC ratios may serve as soil type independent threshold values for SSS.

The quantitative importance of various SOM components on SSS tested using different pretreatments and energy inputs remains elusive. Previous studies often rely on samples retrieved from contrasting sites with different soil types and textures making quantification of the effect of SOM components on SSS dubious.

The objective of this study was to quantify the influence of SOC on several soil structural stability parameters and to test whether SOM characteristics proposed in the literature improve the predictive ability. A wide range of measures for the determination of SSS exists ranging in sample preparation, pretreatment, degree of disturbance and quantification (Le Bissonnais, 1996; Pojasok and Kay, 1990; Pulido Moncada et al., 2015). We applied stability tests varying in pretreatment and ranging from low to very high degree of disturbance focusing on SOC effects on clay dispersibility of 1–2 mm rewetted aggregates, dispersion of particles < 20 µm in < 8 mm field-moist soil, and clay-SOM disintegration of < 2 mm air-dry soil. Soil was from the Highfield Ley-Arable Long-Term Experiment at Rothamsted Research (Highfield-LTE), a silt loam with a wide gradient in SOC. The gradient has developed during at least 56 years of contrasting management practices. We thereby eliminated confounding effects of variations in soil type, soil texture and climate. Soil was from treatments that ensured the widest possible gradient in SOC content and differences in SOM quality.

2. Materials and methods

2.1. The Highfield-LTE and treatments

The Highfield-LTE was established in 1949 on a silt loam soil (Table 1) at Rothamsted Research, Harpenden, UK (51°80'N, 00°36'W) in a field that had been under permanent grass for centuries. The soil belongs to the Batcombe series, and the parent material includes a relatively silty (loess-containing) superficial deposit overlying and mixed with clay-with-flints (Avery and Catt, 1995). The soil is classified as an Aquic Paludalf (USDA Soil Taxonomy System) and Chromic Luvisol (WRB) (Watts and Dexter, 1997). Average annual temperature and precipitation are 10.2 °C (mean of 1992–2014) and 718 mm (mean of 1981–2010), respectively (Scott et al., 2014).

We selected three treatments in the ley-arable experiment:

Continuous arable rotation (A), winter cereals (winter wheat, *Triticum aestivum* L. and winter oats, *Avena sativa* L.) fertilized with 220 kg N ha⁻¹ y⁻¹ and maintained under standard Rothamsted farm

Table 1

Soil characteristics. Within rows, letters denote statistical significance at $P < 0.05$ for the comparison of A, LA and G. An asterisk (*) indicates if BF is significantly different from A, LA and G based on a pairwise t -test. For treatment abbreviations, see Fig. 1.

	BF	A	LA	G
Texture ^a				
Clay < 2 µm	0.270	0.264	0.255	0.261
Silt 2–20 µm	0.249	0.263	0.261	0.272*
Silt 20–63 µm	0.335	0.318	0.324	0.319
Sand 63–2000 µm	0.146	0.155	0.160	0.148
Specific surface area (m ² g ⁻¹ minerals) ^b	56.7	67.9 ^a	68.4 ^a	78.4 ^b
Exchangeable cations and CEC				
Na ⁺ (mmol _c kg ⁻¹ minerals)	0.4	0.5 ^a	0.4 ^a	0.7 ^b
K ⁺ (mmol _c kg ⁻¹ minerals)	3.3	6.3	17.7	5.8
Ca ²⁺ (mmol _c kg ⁻¹ minerals)	93.7	113.3 ^a	128.7 ^a	155.6 ^b
Mg ²⁺ (mmol _c kg ⁻¹ minerals)	5.4	4.0	4.1	4.6
Sum of bases (mmol _c kg ⁻¹ minerals)	102.9	107.1 ^a	120.7 ^{ab}	142.0 ^b
CEC (mmol _c kg ⁻¹ minerals)	145.8	173.8	171.3	209.9*
Base saturation (%)	72.8	65.5	74.8	74.4
pH (CaCl ₂)	5.7	5.1	5.1	5.4

^a kg kg⁻¹ of mineral fraction and based on oven-dry weight.

^b Clay is included as a co-variable as it is significant and makes the treatment effect significant.

practice with straw removed. Ley-arable rotation (LA), three-year grass/clover ley (meadow fescue, *Festuca pratensis* L.; timothy-grass, *Phleum pratense* L.; white clover, *Trifolium repens* L.) followed by three years arable (managed as A). The grass/clover ley received no N fertilizer and the biomass was cut and removed in early summer. The small amount of regrowth was topped in early autumn and left on the plots. Two of the sampled plots were drilled with winter cereals following three years of grass/clover, whereas the other two were drilled with grass/clover following three years of winter cereals. Grass (G), ploughed and reseeded to grass (predominantly rye grass, *Lolium perenne* L.) when the experiment was established (1949). The grass was managed as the grass/clover ley in LA.

We also selected the bare fallow (BF) treatment, which is not part of the original ley-arable experiment but located adjacent to the experiment (denoted Highfield bare fallow and Geescroft bare fallow). The BF treatment has been maintained free of plants by regular tillage (ploughed or rotavated two to four times a year) since 1959. The ploughing depth in BF, A, and LA was 0.23 m. The A, LA and G plots were fertilized with 65 kg P ha⁻¹ and 250 kg K ha⁻¹ every three years.

The A, LA and G treatments were part of a randomized block design with four field replicates, whereas the four BF plots were located at one end of the experiment (Fig. 1). The dimensions of the LA plots were 50 m × 7 m, whereas they were 10 m × 6 m for the other plots. The A, LA and G plots were smaller since they were part of a reversion experiment initiated in 2008. For more details see Johnston (1972) and the electronic Rothamsted Archive (www.era.rothamsted.ac.uk).

2.2. Soil sampling

Soil was sampled in March 2015 at field capacity corresponding approximately to a soil water potential of −100 hPa. Soil blocks (2750 cm³) were carefully retrieved from the 6–15-cm soil layer by use of a spade. Three sampling sites in each experimental plot were randomly chosen and labelled subplot. One block was extracted from each subplot adding up to three blocks per plot. The soil was kept in sturdy containers to prevent soil disturbance during transport and stored in a field-moist condition at 2 °C until required for analyses. Soil from the blocks at subplot level was spread out in steel trays at room temperature, carefully fragmented by hand in several sittings along natural planes of weakness, and finally left to air-dry.

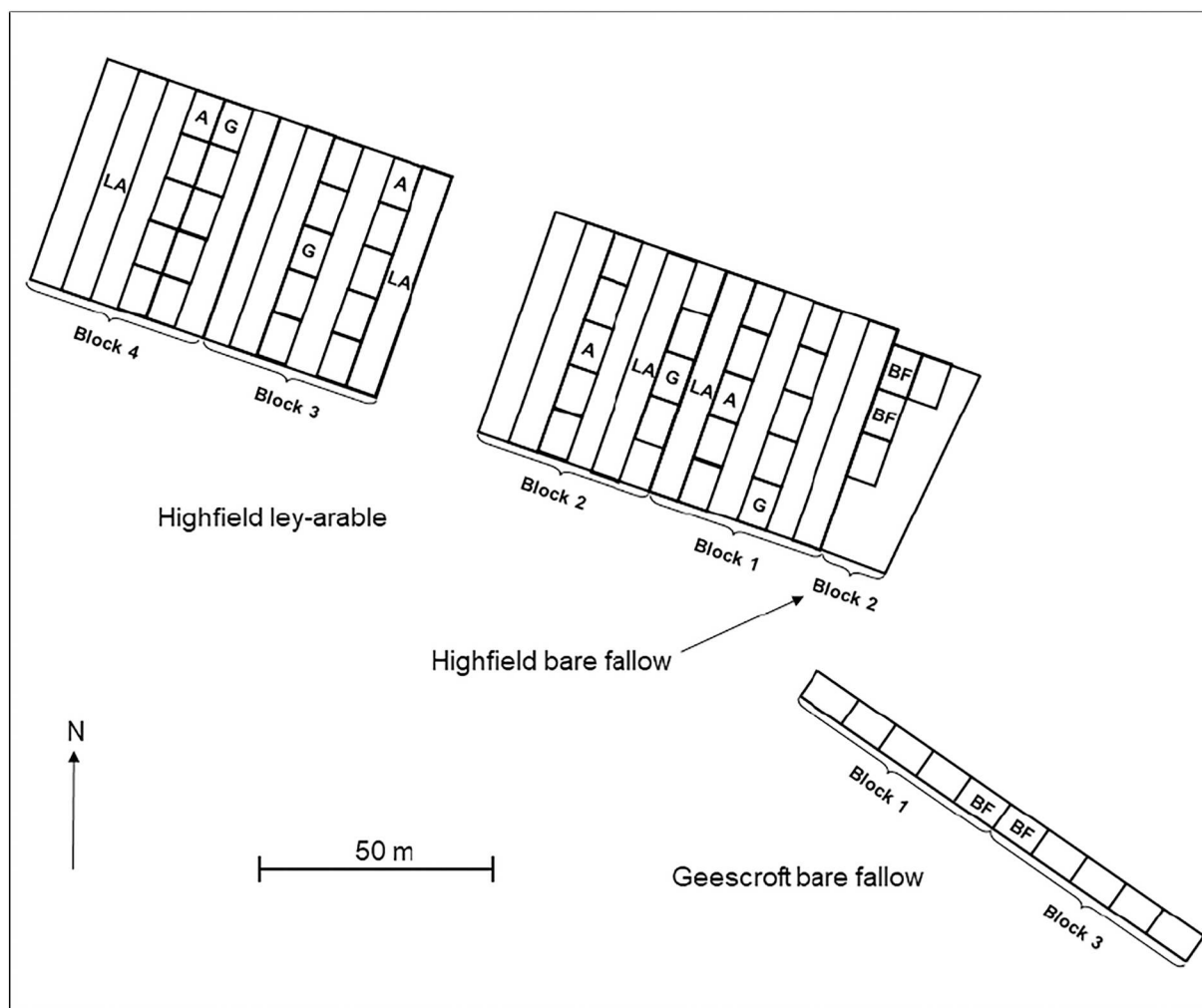


Fig. 1. Distribution of plots in Highfield showing the arable (A), ley-arable (LA) and grass (G) treatments in blocks 1–4 of the ley-arable experiment, and the bare fallow (BF) treatment in blocks 1–3 of the bare fallow experiments.

2.3. Basic chemical and physical analysis

The texture of air-dried bulk soil (crushed and passed through a 2-mm sieve) was determined by the hydrometer method for clay ($< 2 \mu\text{m}$) and silt ($2\text{--}20 \mu\text{m}$) content and the sieve method for mineral particles $> 63 \mu\text{m}$ (Gee and Or, 2002). The soil was tested for CaCO_3 by adding a few droplets of 10% HCl, but none was found. SOM was removed with H_2O_2 before estimation of clay and silt as described in Jensen et al. (2017b). The SOC content was determined on ball-milled subsamples using high-temperature dry combustion (Thermo Flash 2000 NC Soil Analyzer, Thermo Fisher Scientific, Waltham Massachusetts, USA). Specific surface area (SSA) was determined by the ethylene glycol monoethyl ether method (Petersen et al., 1996), and cation exchange capacity (CEC) was determined after Kalra and Maynard (1991). Soil pH was determined in 0.01 M calcium chloride (CaCl_2) solution (1:2.5, w/w). Clay, silt and SOC contents were determined at subplot level, whereas the other properties were determined at plot level.

2.4. Soil organic matter characteristics

Permanganate oxidizable carbon (POXC) was determined at subplot level following Culman et al. (2012). Air-dry 2-mm sieved soil equivalent to 2.5 g oven-dry weight was weighed into a 50 ml falcon tube and shaken end-over-end (33 rpm for 2 min) in 18.0 ml of distilled water and 2.0 ml 0.2 M potassium permanganate (KMnO_4) adjusted to

pH 7.2. After shaking, the soil was allowed to settle for 10 min after which 0.5 ml of the supernatant was transferred to falcon tubes containing 49.5 ml of water. The absorbance of the diluted solution was measured at 550 nm using a spectrophotometer (Thermo Electron Spectronic Helios Alpha Beta UV–visible). The absorbance of four standard stock KMnO_4 solutions was measured to create a standard curve, and sample absorbance was converted to POXC using the equation of Weil et al. (2003).

Hot water-extractable carbon (HWC) was determined at subplot level following Ghani et al. (2003). Briefly, air-dry 2-mm sieved soil equivalent to 3 g oven-dry weight was weighed into a 50 ml falcon tube and shaken end-over-end (33 rpm for 30 min) in 30 ml distilled water at 20°C . After centrifugation (3500 rpm, 20 min) the supernatant was decanted (water soluble carbon), and the soil was re-suspended in distilled water and shaken for 16 h at 200 rpm and 80°C . After centrifugation, the second supernatant (hot water-extractable carbon) was transferred to 50 ml maxi-spin filter tubes equipped with a cellulose acetate membrane filter ($0.45 \mu\text{m}$ pore size) and filtered by centrifugation for 10 min at 3000 rpm. The extracted carbon was determined by wet oxidation using a Shimadzu TOC-V analyzer.

Fractionation based on density was determined at subplot level using a modification of the method described by Sohi et al. (2001). Briefly, 10 g of air-dried 2-mm sieved soil was weighed into a 50 ml falcon tube, 35 ml of NaI solution with a density of 1.8 g cm^{-3} was added, and the solution was shaken at 33 rpm for 2 h. The suspension was centrifuged for 30 min after which floating particles was

Table 2

Soil organic matter characteristics, clay dispersibility of 1–2 mm aggregates rewetted to -100 hPa and clay-SOM disintegration (the ratio between clay content estimated without SOM removal and with removal). Within rows, letters denote statistical significance at $P < 0.05$ for the comparison of A, LA and G. An asterisk (*) indicates if BF is significantly different from A, LA and G based on a pairwise *t*-test. For treatment abbreviations, see Fig. 1.

	BF	A	LA	G
Soil organic matter characteristics				
Soil organic carbon (SOC, g kg ⁻¹ minerals)	9.0	17.3 ^a	21.6 ^a	32.9 ^b
Permanganate oxidizable carbon (POXC, g kg ⁻¹ minerals)	0.161	0.458 ^a	0.600 ^b	0.818 ^c
% of SOC	1.7	2.6 ^{ab}	2.8 ^b	2.5 ^a
Hot water-extractable carbon (HWC, g kg ⁻¹ minerals)	0.437	0.777 ^a	1.082 ^b	1.611 ^c
% of SOC	4.6	4.5 ^a	5.0 ^b	4.9 ^{ab}
Light fraction carbon (LFSOC, g kg ⁻¹ minerals)	0.167	1.285 ^a	1.732 ^a	2.579 ^b
% of SOC	1.9	7.4 [*]	8.0 [*]	7.8 [*]
Aliphatic peak area	58	99 ^a	121 ^a	159 ^b
Aliphatic peak area/SOC	6.5	5.7	5.6	4.9 [*]
Soil structural stability				
Clay dispersibility (DispClay, kg kg ⁻¹ minerals)	0.0115	0.0074 ^c	0.0051 ^b	0.0034 ^a
Clay-SOM disintegration (DI, kg kg ⁻¹ minerals)	1.02	0.96 ^b	1.00 ^b	0.74 ^a

transferred to a glass fiber filter (type GF/A, 110 mm diam., 1.6 μ m retention, Whatman International, Kent, UK), and filtered under suction in a vacuum filtration unit (Büchner funnel). The light fraction organic matter (LFOM) retained on the filter was washed carefully and transferred to a crucible. To ensure a quantitative removal of LFOM the procedure was repeated. The remaining heavy fraction (HF) was washed three times and transferred to a large crucible. The oven-dry weight of the LF and HF was estimated by drying (105 °C for 24 h). The amount of OM recovered was estimated by loss-on-ignition (LOI; 500 °C for 4 h) for both the LFOM and HF. A 5 g air-dry bulk soil sample was dried to allow expressing results on an oven-dry basis after which LOI was determined. The LOI of bulk soil was used to make a model to predict the SOC of the HF based on a multiple regression of SOC against LOI and clay (Model H2.1, Table 2 in Jensen et al., 2018):

$$\text{SOC} = 0.515 \text{ LOI } (P < 0.001) - 0.043 \text{ Clay } (P < 0.001), (n = 48, R^2 = 0.990) \quad (1)$$

Ten tests without soil (blind tests) were performed. The blind test value was subtracted from the LFOM estimate. The LFOM was converted to LFSOC by multiplying with 0.515 (Eq. 1), expressed as percentage of the sum of LFSOC and HFSOC, and normalized to the measured SOC content. Light fraction-free-SOC (LF-free-SOC) was calculated by subtracting LFSOC from SOC.

Fourier transform mid-infrared photoacoustic spectroscopy (FTIR-PAS) was determined at plot level following Peltre et al. (2014). Air-dry 2-mm sieved soil samples were ball-milled and packed in 10-mm diameter cups and the functional groups of soil components recorded using a Nicolet 6700 FTIR spectrometer (Thermo Scientific) equipped with a PA301 photoacoustic detector (Gasera Ltd. Turku, Finland). Spectra were recorded with an average of 32 scans within the range 4000–600 cm⁻¹ and with 2 cm⁻¹ intervals. A flow of helium was used as purge gas to remove noise produced by ambient moisture and CO₂ as well as moisture from the sample after insertion of the cup in the photoacoustic detector chamber. We focused on the 1700–1300 cm⁻¹ region to reduce overlapping bands arising from soil minerals. The spectral peak area between 3000 and 2800 cm⁻¹ was integrated as described in Peltre et al. (2017) and taken to reflect the amount of aliphatics in the soil (Leifeld, 2006).

2.5. Soil structural stability

Clay dispersibility (DispClay) was determined at subplot level on 1–2 mm aggregates isolated from the air-dry 2-mm sieved soil. The aggregates were adjusted to a matric water potential of -100 hPa as described in Schjønning et al. (2012). In short, the aggregates were placed on a tension table at -100 hPa, gradually exposed to reduced suctions until -3 hPa, and finally equilibrated at -100 hPa by gradually increased suctions. The rewetting was with great caution to avoid air explosion (aggregate slaking). Artificial rainwater was added to cylindrical plastic bottles containing 10 g of aggregates in order to obtain a soil:water ratio of 1:8 by weight. After end-over-end rotation (33 rpm, 23-cm diameter rotation) for 2 min, the bottles were left to stand for 230 min, after which the upper 50 mm (60 ml) containing particles $\leq 2 \mu$ m was siphoned off. The weight of dispersed clay was determined after oven-drying (105 °C for 24 h) and corrected for particles $> 250 \mu$ m isolated by chemical dispersion.

Dispersion of particles $< 20 \mu$ m (DispFines20) was measured at different time steps at plot level using field-moist soil. Soil was retrieved with a small corer (22-mm diameter) from the minimally-disturbed soil cubes and gently crumbled by hand to pass an 8-mm sieve. Artificial rainwater was added to a cylindrical bottle containing soil equivalent to 1 g oven-dry weight to obtain a soil:water ratio of 1:100 by weight. The bottle was shaken end-over-end (33 rpm, 23-cm diameter rotation) for 2, 4, 8, 16, 32, 64 and 128 min. At each time step the bottle was left to stand for 67 s, after which the upper 30 ml containing particles $< 20 \mu$ m was siphoned off and the turbidity of the suspension was measured on a Hach 2100AN turbidimeter (Hach, Loveland, CO). After each of the turbidity measurements taken at time steps 2–64 min, the soil suspension was returned to the bottle. Thus, the measurements at the different time steps were on the same sample. After the final measurement (128 min), the 30 ml was transferred to a beaker and bulked at treatment level. For each treatment, correlations between nephelometric turbidity unit (NTU) and particle concentration were established from dilution series. Calibration curves are shown in Fig. S1 in Supplementary material. The results were corrected for particles $> 250 \mu$ m isolated by chemical dispersion.

Soil samples at subplot level were analyzed for clay content without H₂O₂-removal of SOM (Jensen et al., 2017b), and clay-SOM disintegration (DI) was calculated as the ratio between clay content estimated without and with SOM removal. Soil with DI values $< 1 \text{ kg kg}^{-1}$ can be interpreted as being extremely stable since they have resisted disintegration after end-over-end shaking for 18 h in sodium pyrophosphate.

2.6. Calculations and statistics

The soil components measured in this study refer to fractions of oven-dry weight (105 °C for 24 h) of the SOM-free mineral fraction. The components include particle size fractions, SOC, POXC, HWC, LF-free-SOC, SSA, CEC and DispClay. DispFines20 is given as a fraction of SOM-free mineral fraction $< 20 \mu$ m.

For the statistical analysis and processing of spectral data, the R-project software package Version 3.4.0 (R Foundation for Statistical Computing) was used. Treatment effects were analyzed with a linear mixed model including block as a random effect when comparing A, LA and G. The criterion used for statistical significance of treatment effects was $P < 0.05$. When the treatment effect was significant, further analyses were made to isolate differences between treatments (pairwise comparisons) using the general linear hypotheses (*glht*) function implemented in the R *multcomp* package and the Kenward-Roger method to calculate degrees of freedom (Kenward and Roger, 2009). Treatment differences for the comparison of BF and the other treatments were based on a pairwise *t*-test, acknowledging that this is a less robust test, and that the treatment differences could be due to soil variation since the BF treatment is not a part of the ley-arable experiment. Inverse

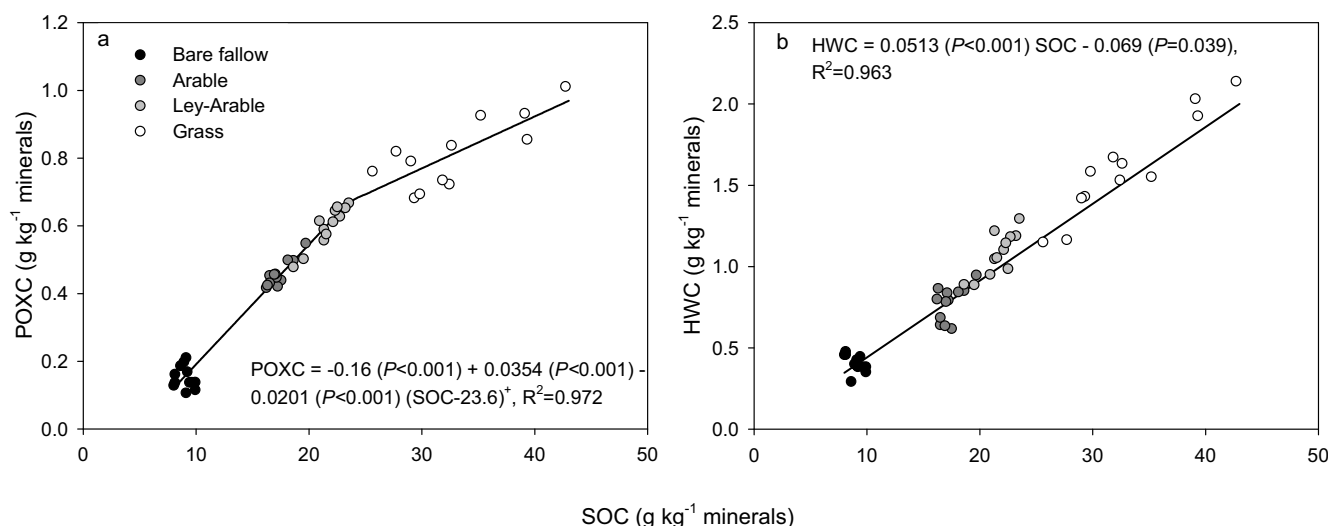


Fig. 2. (a) Permanganate oxidizable carbon (POXC) as a function of SOC and (b) hot water-extractable carbon (HWC) as a function of SOC for the four treatments at subplot level. The broken-stick and linear regression models are indicated.

transformation was performed on DispFines20 to stabilize the variance. The broken-stick model was fitted using the *segmented* package in R. A piece-wise linear model was used:

$$y = \beta_0 + \beta_1(x) + \beta_2(x - c)^+ + e \quad (2)$$

where y is the dependent variable, x is the independent variable, c is the change point and e is the residual standard error (Toms and Lesperance, 2003). The $^+$ sign indicates that the last term only is valid when $x > c$.

Spectral data processing included baseline correction, smoothing using a Savitzky-Golay filter calculated on three data points on each side with a zero-order polynomial, and normalization by the average absorbance of the whole spectra. Principal component analysis (PCA) on the FTIR-PAS spectra was performed using the *ade4* package in R.

3. Results

3.1. Basic soil characteristics

Generally, contents of clay, silt and sand did not differ significantly between treatments (Table 1) allowing the effect of contrasting management to be examined without confounding effects related to soil texture. SSA differed significantly following the same pattern as SOC. CEC was significantly higher for G than for BF treatment, and the amount of exchangeable Ca²⁺ was significantly higher for G compared to other treatments. Soil pH was not affected by the contrasting management practices.

3.2. Soil organic matter characteristics

Concentrations of SOC differed significantly and decreased in the order G > LA = A > BF (Table 2). POXC, HWC, LFSOC, and the aliphatic C-H peak area (3000–2800 cm⁻¹) followed differences in SOC. Supplementary material (Figs. S2 and S3) shows the full spectral range as well as the SOM fingerprint region (1700–1300 cm⁻¹) at plot level.

The aliphatic peak area normalized by SOC content was higher in the BF than in the G treatment indicating that SOM in G soil was depleted in aliphatics. The POXC accounted for 1.7, 2.6, 2.8 and 2.5% of total SOC in BF, A, LA and G treatments, respectively, while HWC contributed 4.6, 4.5, 5.0 and 4.9%. The increase in POXC with an increase in SOC was different for the G treatment compared to A and LA (Fig. 2a). The narrow SOC range in the BF soil did not allow an evaluation of the POXC-SOC relation, but the first slope of the broken-stick

model was similar to a linear regression including A and LA (0.0354 compared to 0.0339 g kg⁻¹ SOC). This indicates that the level of POXC for BF aligned with that of the other tilled treatments (A and LA). The x-intercept of the broken-stick model in Fig. 2a was 4.6 g SOC kg⁻¹ minerals suggesting that no POXC at or below this SOC content was oxidized. For the wide range of SOC in this study, HWC correlated linearly to SOC with an intercept value close to zero (Fig. 2b). Our data thus indicates that HWC represents a fixed proportion of SOC (~0.05 g HWC per g SOC or ~5%).

The PCA analysis based on the 1700–1300 cm⁻¹ region clearly separated the treatments on the first principal component (PC1) explaining 84.6% of the spectral variance (Fig. 3a). Field plots from the G and BF treatment were located to the left and right side of the PCA plane, respectively. The A and LA treatments were in the center of the PCA plane and did not differ much. The loading of PC1 indicated that BF soils were enriched in organic compounds vibrating between 1700 and 1580 cm⁻¹ with a peak at 1625 cm⁻¹ (Fig. 3b). Absorption in this region is attributed aromatics and carboxylate at 1600–1570 cm⁻¹, amine at 1610 cm⁻¹, clay-bound water at 1640 cm⁻¹, aromatics at 1660–1600 cm⁻¹ and amides at 1670–1640 cm⁻¹ (Table 1 in Peltre et al., 2017). In contrast, G soils were relatively enriched in organic compounds vibrating between 1580 and 1495 cm⁻¹ and 1475–1325 cm⁻¹ with peaks at 1550, 1510 and 1385 cm⁻¹ (Fig. 3b). These regions are attributed nitrate at 1380 cm⁻¹, carboxylate at 1390 cm⁻¹, amide III at 1420 cm⁻¹, carbonates at 1430 cm⁻¹, aliphatic methyls at 1445–1350 cm⁻¹, lignin rings at 1505–1515 cm⁻¹ and amide II at 1570–1540 cm⁻¹ (Table 1 in Peltre et al., 2017).

3.3. Soil structural stability

The amount of dispersible clay differed significantly, decreasing in the order BF > A > LA > G, and the disintegration of soil without SOM removal was significantly lower for the G treatment compared to the other treatments (Table 2). Linear, semi-logarithmic and broken-stick models were employed to describe the correlations of SOC, POXC and HWC to DispClay and DI (Table 3). The coefficient of determination (R²) peaked when DispClay and DI were related to SOC with a broken-stick model (see relation on Fig. 4a). Similar relationships were found when relating POXC and HWC to DispClay and DI (Fig. 4b and c).

Relating changes in DispClay to LF-free-SOC did not improve R² compared to SOC (Fig. 5a), whereas LF-free-SOC increased the explained variation in DI by 1%-unit (Fig. 5b).

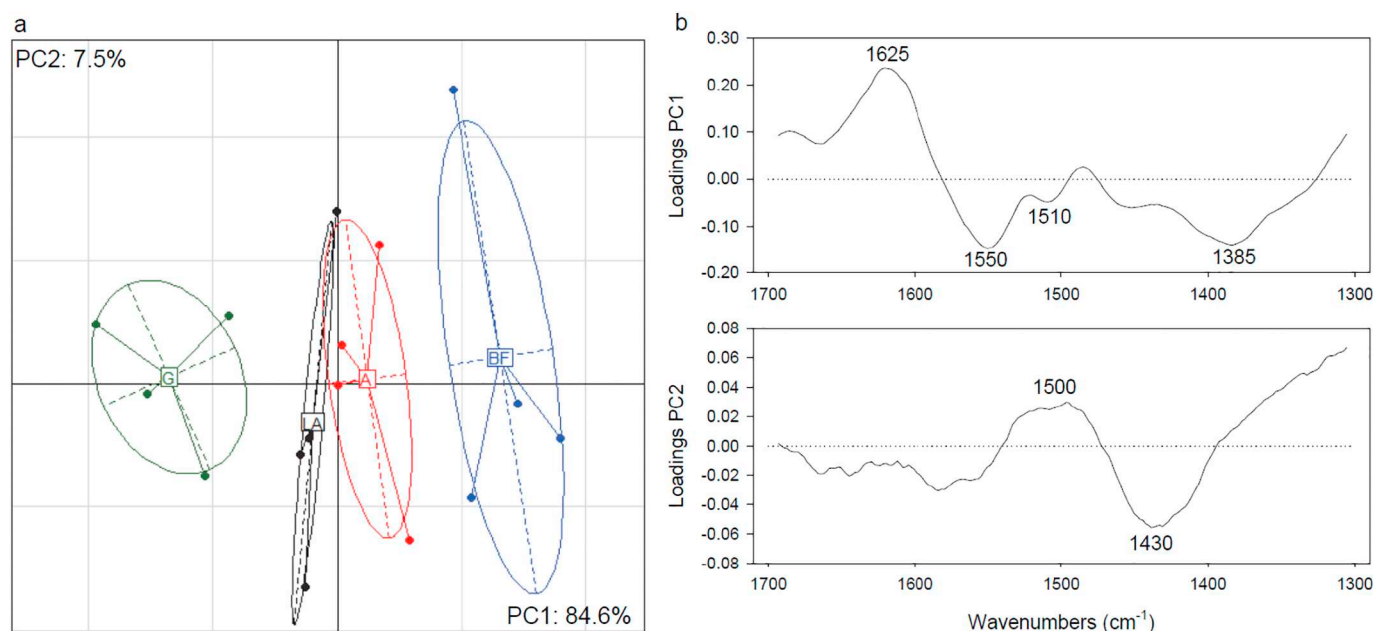


Fig. 3. Principal component analysis (PCA) based on FTIR-PAS spectra for the different treatments. The dots indicate the four plots of each treatment. For treatment abbreviations, see Fig. 1. (a) Scores plot in the plane defined by principal component 1 (PC1, explaining 84.6% of the variance) and principal component 2 (PC2, explaining 7.5% of the variance) of the PCA. (b) Loadings of the PCA for PC1 and PC2.

DispFines20 was significantly lower for the G treatment compared to the other treatments at every time step (Fig. 6a), and the release-curve had a contrasting shape compared to that of the other treatments. DispFines20 was significantly lower for LA than for A treatment after both 64 and 128 min. The release rate was markedly higher in the beginning for BF, A and LA compared to G (Fig. 6b). After c. 24 min, G showed a higher release rate than the other treatments. At every step, DispFines20 was virtually constant across the four G treatment plots despite a range in SOC, while considerable variation was observed for the narrower SOC ranges of the other three treatments (Fig. S4 in Supplementary material).

4. Discussion

4.1. Linking soil organic matter components to soil structural stability

POXC has been advocated as a partly processed but reactive pool of SOM (Culman et al., 2012), able to support biological functions (Idowu et al., 2008), while HWC has been promoted as an indicator of soil biochemical quality (Ghani et al., 2003). Both fractions are supposedly sensitive indicators for the assessment of management-induced changes in SOM quality (Culman et al., 2012; Ghani et al., 2003). Labile organic compounds are known to bond primary mineral particles into differently

Table 3

Parameters of the linear, semi-logarithmic and broken-stick models for clay dispersibility (DispClay; kg kg⁻¹ minerals) and clay-SOM disintegration (DI; kg kg⁻¹ minerals) as a function of soil organic carbon (SOC; g kg⁻¹ minerals), permanganate oxidizable carbon (POXC; g kg⁻¹ minerals), and hot water-extractable carbon (HWC; g kg⁻¹ minerals). The change point of the broken-stick model and the corresponding 95% confidence interval is indicated. The relation between the first and second slope estimate of the broken-stick model (Slope1/Slope2) was calculated if both slopes were significant. The coefficient of determination (R²) is indicated.

Predictor	Model		Equation	Change point	Slope1/Slope2	R ²
SOC	Linear	DispClay	0.0134*** – 0.00032*** SOC			0.839
		DI	1.16*** – 0.0116*** SOC			0.723
	Semi-log	DispClay	0.0255*** – 0.0148*** log(SOC)			0.930
		DI	1.49*** – 0.442*** log(SOC)			0.555
	Broken-stick	DispClay	0.0160*** – 0.00049*** SOC + 0.00039*** (SOC-23.5) ⁺	23.5*** [20.9;26.0]	4.6	0.940
		DI	1.03*** – 0.0029 ^{NS} SOC – 0.018*** (SOC-22.5) ⁺	22.5*** [19.9;25.1]		0.880
POXC	Linear	DispClay	0.0131*** – 0.0122*** POXC			0.907
		DI	1.11*** – 0.366*** POXC			0.550
	Semi-log	DispClay	0.0029*** – 0.011*** log(POXC)			0.891
		DI	0.83*** – 0.261*** log(POXC)			0.364
	Broken-stick	DispClay	0.0136*** – 0.0138*** POXC + 0.0084* (POXC-0.694) ⁺	0.694* [0.564;0.824]	2.6	0.927
		DI	1.02*** – 0.087 ^{NS} POXC – 1.00*** (POXC-0.628) ⁺	0.628*** [0.573;0.683]		0.819
HWC	Linear	DispClay	0.0128*** – 0.0061*** HWC			0.815
		DI	1.13*** – 0.21*** HWC			0.648
	Semi-log	DispClay	0.0059*** – 0.0133*** log(HWC)			0.900
		DI	0.90*** – 0.378*** log(HWC)			0.490
	Broken-stick	DispClay	0.0156*** – 0.0105*** HWC + 0.00760*** (HWC-0.970) ⁺	0.970*** [0.833;1.107]	3.6	0.913
		DI	1.02*** – 0.049 ^{NS} HWC – 0.341*** (HWC-1.104) ⁺	1.104*** [0.921;1.288]		0.788

NS: Not significant.

* Indicates significance level at $P < 0.05$.

*** Indicates significance level at $P < 0.001$.

⁺ Indicates that the last term is valid only when the content of SOC, POXC or HWC are larger than the change point.

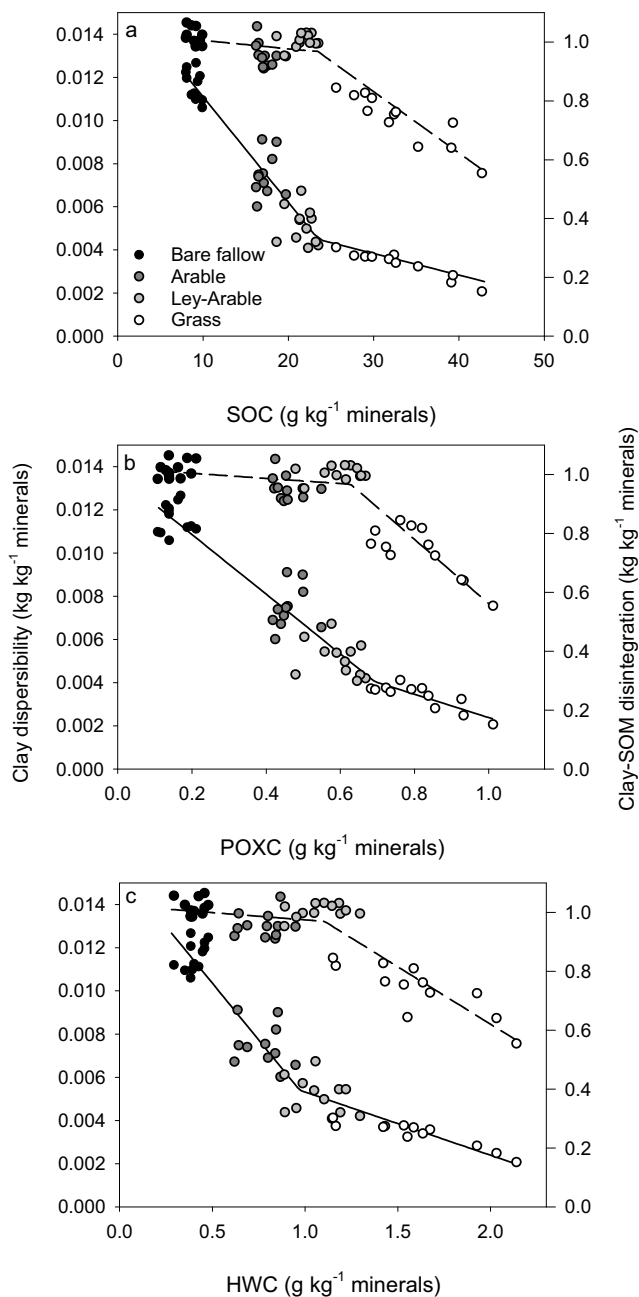


Fig. 4. Clay dispersibility of 1–2 mm aggregates rewetted to -100 hPa (solid lines) and disintegration (the ratio between clay content estimated without SOM removal and with removal) (dashed lines) as a function of (a) soil organic carbon (SOC), (b) permanganate oxidizable carbon (POXC), and (c) hot water-extractable carbon (HWC) for the four treatments at subplot level. The broken-stick models (Table 3) are indicated. See Table 3 for equations and R^2 -values.

sized organo-mineral complexes and stabilize these against mechanical damage (Degens, 1997). However, the clay-SOM disintegration (DI) test involves extreme disruptive energy (end-over-end shaking for 18 h in sodium pyrophosphate solution) and is not expected to be affected by variations in labile SOC components. In accordance with this, we noted a higher coefficient of determination in the broken-stick regression relating DI to SOC ($R^2 = 0.88$) than in those for POXC and HWC ($R^2 = 0.82$ and $R^2 = 0.79$, respectively). One may speculate that stable organo-mineral associations (i.e. at sub-micro-aggregate scale) are responsible for the extreme stability at high SOC contents. The similar pattern observed for POXC and HWC (broken-stick) relates to the near linear relations observed between total SOC and these two fractions (Fig. 2).

For the DispClay measure of SSS, we observed nearly identical coefficients of determination in the broken-stick models describing the data: R^2 equals 0.94, 0.93 and 0.91 with SOC, POXC and HWC as predictor (Table 3). We further noted that the broken-stick was “less broken” especially when using POXC as predictor (slope ratios, Table 3). This observation is supported by the higher ability of POXC to describe data in a linear model ($R^2 = 0.91$) compared to HWC and SOC ($R^2 = 0.82$ and $R^2 = 0.84$, respectively). Overall, this may indicate that POXC is superior to SOC and HWC in describing the variation in DispClay.

Our study does not reveal mechanisms leading to SSS. The indication that POXC is superior in describing the variation in DispClay suggests a link to bonding agents such as polysaccharides, assumed to be important bonding agents at micro-aggregate scale (Tisdall and Oades, 1982). However, the composition of POXC remains unknown as it is destroyed by oxidation. The lower predictive ability of HWC to explain SSS data and its close correlation to SOC may indicate that it is a less relevant quality characteristic of SOC. Other studies have emphasized that hot water-extractable carbohydrate-C is a better predictor of SSS than SOC (Haynes, 2005; Haynes and Swift, 1990).

Changes in DispClay and DI may relate better to LF-free-SOC than total SOC. However, the difference in using LF-free-SOC compared to SOC was marginal (Fig. 5). The decrease in DispClay and DI when going from BF to G may relate to an enrichment in aliphatics and lignin, and a decrease in carboxylic groups and amides (Table 2, Fig. 3). The higher amount of carboxyls and amides in BF soils suggest that SOM is more oxidized in these soils. This agrees with the findings of Barré et al. (2016), and carboxyls and amides may be related to microbial processed and stable SOM in organo-mineral associations (Kleber et al., 2015). The higher proportion of aliphatics in SOM from the BF soils also supports the presence of a more decomposed SOM. The results indicate that plant residues, initially present in the BF soil, decomposed rapidly leaving behind SOM enriched in microbial-derived OM. In contrast, less oxidized plant residues accounts for a larger fraction of the SOM in the G soils.

4.2. Management system effects on soil structural stability

The four treatments reflect three management systems comprising the BF treatment with intensive tillage, no plants or carbon input, the A and LA treatments with plants and tillage, and the G treatment with plants and absence of tillage. The G treatment differed from the other treatments by having a very stable structure and a better ability to resist physical disturbance. Consequently, the potential maximum SSS was reached only in the long-term G treatment. This was reflected in the DispClay and DI tests, where the change point of the broken-stick model for SOC content was confounded with treatment and represented a change from the LA to G treatment (Fig. 4a). Also, the results on DispFines20 support a difference in stability between the G treatment and the other treatments illustrated by the contrasting release-curve, release rate and higher stability at every time step (Fig. 6). Permanent grass stands out from the other treatments by having a greater input of above- and belowground plant residues. Hirsch et al. (2009, 2017) found markedly greater numbers of roots and mesofauna in G compared to BF and A, and a larger abundance of fungi. Roots and fungal hyphae can act as binding agents, enmeshing aggregates (Elmholt et al., 2008; Tisdall and Oades, 1982), and potentially increasing SSS, while mesofauna fecal pellets can contribute by stabilizing decomposition products (Oades, 1993). In addition, the effects of these stabilizing agents persist since they are continuously replaced and the resulting structure remains undisturbed due to the absence of tillage. The limited effect of SOC on DispFines20 within the G treatment at every time step (Fig. S4 in Supplementary material) may relate to the larger scale applied in the test, i.e. whole-soil samples not broken down to more than 8 mm. At this scale, management derived drivers such as macro-aggregate stabilizing agents appear more important, while SOC may play a minor

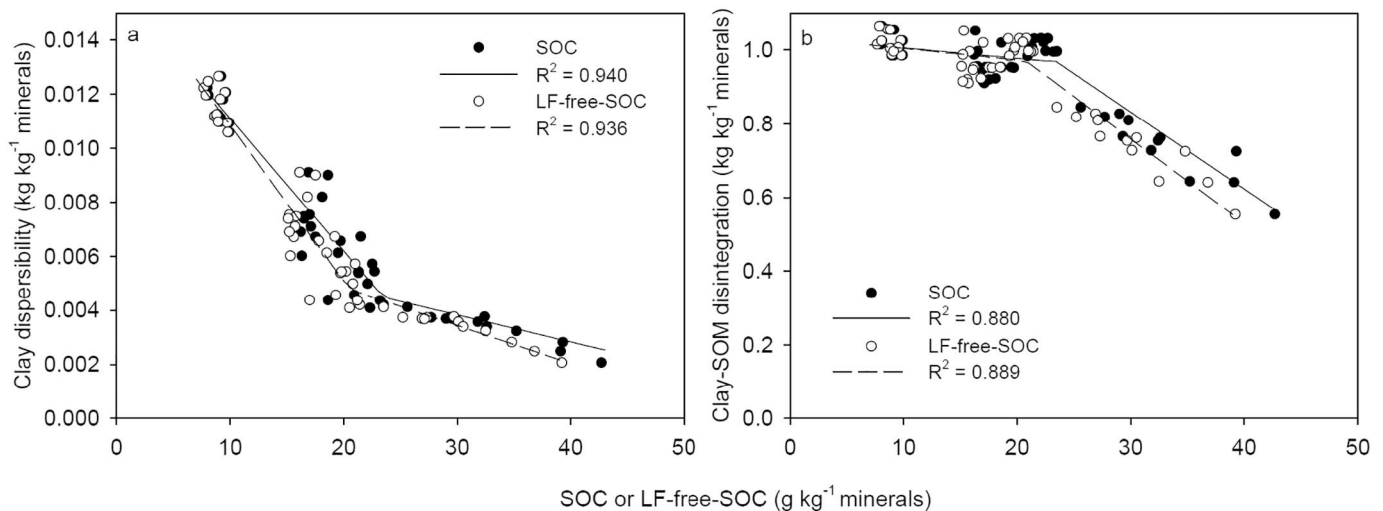


Fig. 5. (a) Clay dispersibility and (b) Clay-SOM disintegration as a function of soil organic carbon (SOC; black symbols) and LF-free-SOC (Light fraction-free-SOC; white symbols). The broken-stick models and R²-values are indicated.

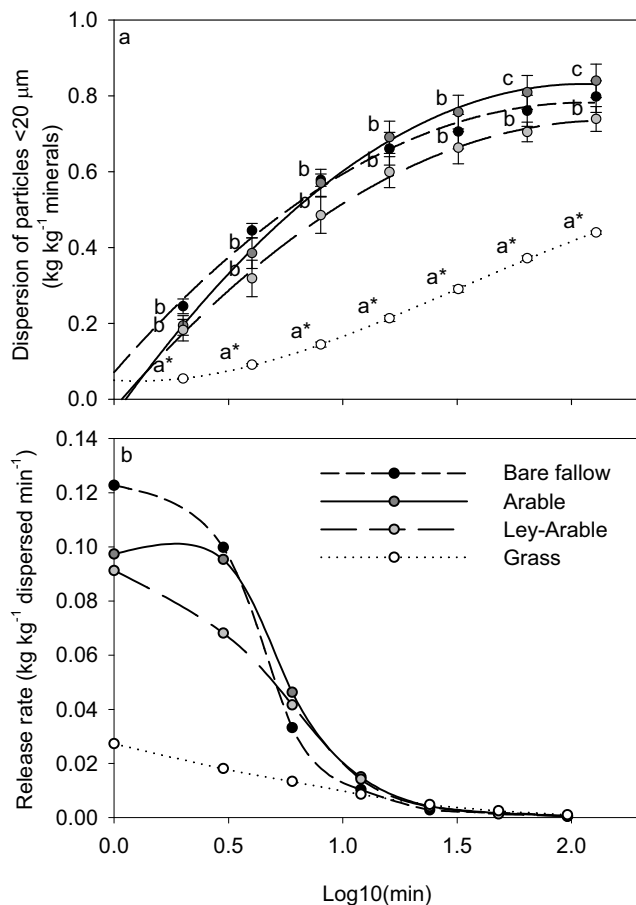


Fig. 6. (a) The ratio between dispersed particles < 20 μm and the total content of particles < 20 μm as a function of Log10(min) at treatment level. The standard error of the mean is indicated ($n = 4$). The polynomials are fitted to the four replicates time's seven data points per treatment. Letters denote statistical significance at $P < 0.05$ for the comparison of A, LA and G. An asterisk (*) indicates if BF is significantly different from A, LA and G based on a pairwise t -test. (b) Release rate (kg kg⁻¹ minerals dispersed min⁻¹) as a function of Log10(min) at treatment level. A stepwise, simple calculation of slope from time step to time step was employed to calculate the release rate, and a smoothed spline curve was added.

role. The greater stability of the G treatment may also link to the absence of tillage, known to be detrimental to the preservation of stabilizing agents.

The higher concentration of soluble Ca²⁺ ions in the G treatment may promote flocculation of clay particles (Le Bissonnais, 1996) contributing to the higher structural stability. However, the importance of cations for aggregate stability is considered less important in soils high in clay or SOC (Bronick and Lal, 2005). Matthews et al. (2008) found a decrease in wettability for G, whereas wettability was similar for BF, A and LA treatments. Thus, decreased wettability may contribute to the very stable structure of the G soil.

4.3. Critical carbon levels

Our SSS measures DI and DispClay showed a change in the relation to SOC at around 23.0 g kg⁻¹ minerals (broken-stick change point; Fig. 4a). The carbon saturation concept (Six et al., 2002; Stewart et al., 2007) implies the existence of a SOC concentration that for a given soil provides a full “coverage” of the surface of soil minerals with SOC. This concept of a finite SOC storage capacity (Ingram and Fernandes, 2001) was supported by results for a range of grassland soils assumed to be saturated with SOC (Hassink, 1997). Thus, the SOC concentration of ~23.0 g kg⁻¹ minerals found in this study may reflect a potential storage capacity for this soil. The broken-stick pattern for DispClay indicates that SOC influences SSS more for soils with SOC below the change point than above (Fig. 4a). DispClay increases more with reduction in SOC when the soil is unsaturated (below the change point) than when it is saturated. This is in line with Jensen et al. (2017a), who found SOC to be important for SSS in SOM-depleted soil. Interestingly, the DI test with extreme energy input showed that all unsaturated soil samples behaved similarly and showed complete disintegration (slope not significantly different from zero; Fig. 4a).

The threshold for a change in SOC effects on SSS differ from soil to soil, e.g. around 11.0 g SOC kg⁻¹ minerals for a sandy loam in Denmark (Jensen et al., 2017a). Thus, the SOC level critical to SSS seems soil type dependent. Dexter et al. (2008) and Jensen et al. (2017a) found increasingly compromised SSS when the clay/SOC ratio was above 10. Schjøning et al. (2012) and Jensen et al. (2017a) found that a Fines20/SOC ratio of 20 serve as a similar critical threshold value. The clay/SOC and Fines20/SOC ratios for a change in DispClay and DI were calculated by dividing the average clay or Fines20 content with the change point, giving values of 11 and 23, respectively. Thus, our results support the soil clay/SOC~10 or Fines20/SOC~20 as defining factors for SSS.

In this study, the thresholds for changes in SOC (as well as POXC and HWC) effects on SSS are confounded with management (Fig. 4). Thus the calculated soil mineral fines/SOC thresholds may relate to a quantity of SOC as well as to management system (as discussed in Section 4.2). This was unavoidable since creating systems with a wide range in SOC often involves the use of contrasting management. However, confounding effects derived from differences in soil type, soil texture and climate were eliminated in our study. We encourage further studies on soils with a gradient in SOC and little variation in texture, and where the mineral fines/SOC thresholds do not coincide with a shift in management system.

5. Conclusions

We exploited the unique range in SOM within the Highfield-LTE developed from contrasting long-term managements. Soil structural stability (SSS) increased with an increase in SOM components. However, the relationships followed a broken-stick regression with the greater effect occurring when SOM components were low. The SOM fractions permanganate oxidizable carbon (POXC) and hot water-extractable carbon (HWC) were less related to clay-SOM disintegration than was SOC. However, POXC seemed superior in describing the variation in clay dispersibility compared to SOC and HWC. The permanent grass had a very stable structure, even when exposed to a high degree of disturbance. This may relate to the management system supporting large concentrations of stabilizing agents due to large annual inputs of above- and belowground plant residues as well as the absence of tillage. This management system facilitates high abundance of soil microbiota and mesofauna. For the present soil, SOM promoting management increased SSS up to a specific threshold coinciding with a change to permanent grass. For soils low in SOC, increasing the SOC content or changing the management from arable rotation to permanent grass improve SSS. Further, this study supports the existence of critical soil mineral fines/SOC ratios for SSS with change points at clay/SOC~10 and Fines20/SOC~20.

Acknowledgements

We gratefully acknowledge the technical assistance of Stig T. Rasmussen, Dept. Agroecology (Aarhus University), and the technical staff at Rothamsted Research. We thank Bodil B. Christensen, Palle Jørgensen, Karin Dyrberg, Kim M. Johansen, and Morgane Coulumbel for technical assistance. We thank Kristian Kristensen for statistical advice. The study was supported by the Green Development and Demonstration Programme (GUDP) of the Ministry of Environment and Food of Denmark through the “Cover crops for optimization of cereal based cropping systems” (Grant No. 3405-11-0225) and “Optimized soil tillage in cereal based cropping systems” (Grant No. 34009-12-0502) projects, and by the EU 7th Research Framework Programme, Distributed Infrastructure for Experimentation in Ecosystem Research (ExpeER) through the project “Identification of soil organic carbon thresholds for sustained soil functions in agroecosystems” (Grant No. 262060). The Rothamsted Long-term Experiments National Capability (grant code BBS/E/C00J0300) is supported by the UK Biotechnology and Biological Sciences Research Council (BBSRC) and the Lawes Agricultural Trust.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geoderma.2018.10.034>.

References

Amundson, R., Berhe, A.A., Hopmans, J.W., Olson, C., Sztein, A.E., Sparks, D.L., 2015. Soil and human security in the 21st century. *Science* 348, 1261071.

- Avery, B.W., Catt, J.A., 1995. The Soils at Rothamsted. Lawes Agricultural Trust, pp. 1–44.
- Barré, P., Plante, A.F., Cécillon, L., Lutfalla, S., Baudin, F., Bernard, S., Christensen, B.T., Eglin, T., Fernandez, J.M., Houot, S., Kätterer, T., Le Guillou, C., Macdonald, A., van Oort, F., Chenu, C., 2016. The energetic and chemical signatures of persistent soil organic matter. *Biogeochemistry* 130, 1–12.
- Bronick, C.J., Lal, R., 2005. Soil structure and management: a review. *Geoderma* 124, 3–22.
- Culman, S.W., Snapp, S.S., Freeman, M.A., Schipanski, M.E., Beniston, J., Lal, R., Drinkwater, L.E., Franzluebbers, A.J., Glover, J.D., Grandy, A.S., Lee, J., Six, J., Maul, J.E., Mirsky, S.B., Spargo, J.T., Wander, M.M., 2012. Permanganate oxidizable carbon reflects a processed soil fraction that is sensitive to management. *Soil Sci. Soc. Am. J.* 76, 494–504.
- de Jonge, L.W., Kjaergaard, C., Moldrup, P., 2004. Colloids and colloid-facilitated transport of contaminants in soils. *Vadose Zone J.* 3, 321–325.
- Degens, B.P., 1997. Macro-aggregation of soils by biological bonding and binding mechanisms and the factors affecting these: a review. *Aust. J. Soil Res.* 35, 431–460.
- Dexter, A.R., 1988. Advances in characterization of soil structure. *Soil Tillage Res.* 11, 199–238.
- Dexter, A., Richard, G., Arrouays, D., Czyż, E., Jolivet, C., Duval, O., 2008. Complexed organic matter controls soil physical properties. *Geoderma* 144, 620–627.
- Elmholt, S., Schjønning, P., Munkholm, L.J., Debosz, K., 2008. Soil management effects on aggregate stability and biological binding. *Geoderma* 144, 455–467.
- Fine, A.K., van Es, H.M., Schindelfbeck, R.R., 2017. Statistics, scoring functions, and regional analysis of a comprehensive soil health database. *Soil Sci. Soc. Am. J.* 81, 589–601.
- Gee, G.W., Or, D., 2002. Particle-size analysis. In: Dane, J.H., Topp, G.C. (Eds.), *Methods of Soil Analysis. Part 4 - Physical Methods*. Soil Science Society of America, Inc., Madison, Wisconsin, USA, pp. 255–294.
- Getahun, G.T., Munkholm, L.J., Schjønning, P., 2016. The influence of clay-to-carbon ratio on soil physical properties in a humid sandy loam soil with contrasting tillage and residue management. *Geoderma* 264 (Part A), 94–102.
- Ghani, A., Dexter, M., Perrott, K.W., 2003. Hot-water extractable carbon in soils: a sensitive measurement for determining impacts of fertilisation, grazing and cultivation. *Soil Biol. Biochem.* 35, 1231–1243.
- Gregorich, E.G., Beare, M.H., McKim, U.F., Skjemstad, J.O., 2006. Chemical and biological characteristics of physically uncomplexed organic matter. *Soil Sci. Soc. Am. J.* 70, 975–985.
- Hassink, J., 1997. The capacity of soils to preserve organic C and N by their association with clay and silt particles. *Plant Soil* 191, 77–87.
- Haynes, R.J., 2005. Labile organic matter fractions as central components of the quality of agricultural soils: an overview. *Adv. Agron.* 85, 221–268.
- Haynes, R.J., Swift, R.S., 1990. Stability of soil aggregates in relation to organic constituents and soil water content. *J. Soil Sci.* 41, 73–83.
- Hirsch, P.R., Gilliam, L.M., Sohi, S.P., Williams, J.K., Clark, I.M., Murray, P.J., 2009. Starving the soil of plant inputs for 50 years reduces abundance but not diversity of soil bacterial communities. *Soil Biol. Biochem.* 41, 2021–2024.
- Hirsch, P.R., Jhurrea, D., Williams, J.K., Murray, P.J., Scott, T., Misselbrook, T.H., Goulding, K.W.T., Clark, I.M., 2017. Soil resilience and recovery: rapid community responses to management changes. *Plant Soil* 412, 283–297.
- Hurisso, T.T., Culman, S.W., Horwath, W.R., Wade, J., Cass, D., Beniston, J.W., Bowles, T.M., Grandy, A.S., Franzluebbers, A.J., Schipanski, M.E., Lucas, S.T., Ugarte, C.M., 2016. Comparison of permanganate-oxidizable carbon and mineralizable carbon for assessment of organic matter stabilization and mineralization. *Soil Sci. Soc. Am. J.* 80, 1352–1364.
- Idowu, O.J., van Es, H.M., Abawi, G.S., Wolfe, D.W., Ball, J.I., Gugino, B.K., Moebius, B.N., Schindelfbeck, R.R., Bilgili, A.V., 2008. Farmer-oriented assessment of soil quality using field, laboratory, and VNIR spectroscopy methods. *Plant Soil* 307, 243–253.
- Ingram, J.S.I., Fernandes, E.C.M., 2001. Managing carbon sequestration in soils: concepts and terminology. *Agric. Ecosyst. Environ.* 87, 111–117.
- Jensen, J.L., Schjønning, P., Christensen, B.T., Munkholm, L.J., 2017a. Suboptimal fertilisation compromises soil physical properties of a hard-setting sandy loam. *Soil Res.* 55, 332–340.
- Jensen, J.L., Schjønning, P., Watts, C.W., Christensen, B.T., Munkholm, L.J., 2017b. Soil texture analysis revisited: removal of organic matter matters more than ever. *PLoS One* 12, e0178039.
- Jensen, J.L., Christensen, B.T., Schjønning, P., Watts, C.W., Munkholm, L.J., 2018. Converting loss-on-ignition to organic carbon content in arable topsoil: pitfalls and proposed procedure. *Eur. J. Soil Sci.* <https://doi.org/10.1111/ejss.12558>.
- Johnston, A.E., 1972. The effect of ley and arable cropping systems on the amount of soil organic matter in Rothamsted and Woburn Ley-Arable experiments. In: Report Rothamsted Experimental Station for 1972, Part 2, pp. 131–152.
- Johnston, A.E., Poulton, P.R., Coleman, K., 2009. Chapter 1 soil organic matter: its importance in sustainable agriculture and carbon dioxide fluxes. *Adv. Agron.* 101, 1–57.
- Kalra, Y.P., Maynard, D.G., 1991. *Methods Manual for Forest Soil and Plant Analysis*. Northern Forestry Centre, Edmonton, Alberta.
- Kay, B.D., Munkholm, L.J., 2004. Management-induced soil structure degradation - organic matter depletion and tillage. In: Schjønning, P., Elmholt, S., Christensen, B.T. (Eds.), *Managing Soil Quality: Challenges in Modern Agriculture*. CABI Publishing, Wallingford, UK, pp. 185–197.
- Kenward, M.G., Roger, J.H., 2009. An improved approximation to the precision of fixed effects from restricted maximum likelihood. *Comput. Stat. Data Anal.* 53, 2583–2595.
- Kleber, M., Eusterhues, K., Keiluweit, M., Mikutta, C., Mikutta, R., Nico, P.S., 2015. Mineral-organic associations: formation, properties and relevance in soil environments. *Adv. Agron.* 130, 1–140.

- Le Bissonnais, Y., 1996. Aggregate stability and assessment of soil crustability and erodibility: I theory and methodology. *Eur. J. Soil Sci.* 47, 425–437.
- Leifeld, J., 2006. Application of diffuse reflectance FT-IR spectroscopy and partial least-squares regression to predict NMR properties of soil organic matter. *Eur. J. Soil Sci.* 57, 846–857.
- Matthews, G.P., Watts, C.W., Powlson, D.S., Price, J.C., Whalley, W.R., 2008. Wetting of agricultural soil measured by a simplified capillary rise technique. *Eur. J. Soil Sci.* 59, 817–823.
- McNally, S.R., Beare, M.H., Curtin, D., Meenken, E.D., Kelliher, F.M., Calvelo Pereira, R., Shen, Q., Baldock, J., 2017. Soil carbon sequestration potential of permanent pasture and continuous cropping soils in New Zealand. *Glob. Chang. Biol.* 23, 4544–4555.
- Oades, J.M., 1993. The role of biology in the formation, stabilization and degradation of soil structure. *Geoderma* 56, 377–400.
- Peltre, C., Bruun, S., Du, C., Thomsen, I.K., Jensen, L.S., 2014. Assessing soil constituents and labile soil organic carbon by mid-infrared photoacoustic spectroscopy. *Soil Biol. Biochem.* 77, 41–50.
- Peltre, C., Gregorich, E.G., Bruun, S., Jensen, L.S., Magid, J., 2017. Repeated application of organic waste affects soil organic matter composition: evidence from thermal analysis, FTIR-PAS, amino sugars and lignin biomarkers. *Soil Biol. Biochem.* 104, 117–127.
- Petersen, L.W., Moldrup, P., Jacobsen, O.H., Rolston, D.E., 1996. Relations between specific surface area and soil physical and chemical properties. *Soil Sci.* 161, 9–21.
- Pojasok, T., Kay, B.D., 1990. Assessment of a combination of wet sieving and turbidimetry to characterize the structural stability of moist aggregates. *Can. J. Soil Sci.* 70, 33–42.
- Pulido Moncada, M., Gabriels, D., Cornelis, W., Lobo, D., 2015. Comparing aggregate stability tests for soil physical quality indicators. *Land Degrad. Dev.* 26, 843–852.
- Schjønning, P., de Jonge, L.W., Munkholm, L.J., Moldrup, P., Christensen, B.T., Olesen, J.E., 2012. Clay dispersibility and soil friability—testing the soil clay-to-carbon saturation concept. *Vadose Zone J.* 11, 174–187.
- Scott, T., Macdonald, A.J., Goulding, K.W.T., 2014. The UK Environmental Change Network, Rothamsted. Physical and Atmospheric Measurements: The First 20 Years. Lawes Agricultural Trust Co. Ltd., Harpenden.
- Six, J., Conant, R.T., Paul, E.A., Paustian, K., 2002. Stabilization mechanisms of soil organic matter: implications for C-saturation of soils. *Plant Soil* 241, 155–176.
- Sohi, S.P., Mahieu, N., Arah, J.R.M., Powlson, D.S., Madari, B., Gaunt, J.L., 2001. A procedure for isolating soil organic matter fractions suitable for modeling. *Soil Sci. Soc. Am. J.* 65, 1121–1128.
- Stewart, C.E., Paustian, K., Conant, R.T., Plante, A.F., Six, J., 2007. Soil carbon saturation: concept, evidence and evaluation. *Biogeochemistry* 86, 19–31.
- Tisdall, J.M., Oades, J.M., 1982. Organic matter and water-stable aggregates in soils. *J. Soil Sci.* 33, 141–163.
- Toms, J.D., Lesperance, M.L., 2003. Piecewise regression: a tool for identifying ecological thresholds. *Ecology* 84, 2034–2041.
- Villada, A., Vanguelova, E.I., Verhoef, A., Shaw, L.J., 2016. Effect of air-drying pre-treatment on the characterization of forest soil carbon pools. *Geoderma* 265 (Supplement C), 53–61.
- Watts, C.W., Dexter, A.R., 1997. The influence of organic matter in reducing the destabilization of soil by simulated tillage. *Soil Tillage Res.* 42, 253–275.
- Weil, R.R., Islam, K.R., Stine, M.A., Gruver, J.B., Samson-Liebig, S.E., 2003. Estimating active carbon for soil quality assessment: a simplified method for laboratory and field use. *Am. J. Altern. Agric.* 18, 3–17.